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IN THE UNITED STATES PATENT & TRADEMARK OFFICE

IN RE APPLICATION OF	:	
KENSAKU KOMATSU, ET AL.	:	EXAMINER : FORTUNA, ANA
SERIAL NO: 09/884,084	:	
FILES: JUNE 20, 2001	:	GROUP ART UNIT: 1723
FOR: POROUS HOLLOW FIBER MEMBRANES AND METHOD OF MAKING THE SAME	:	

DECLARATION UNDER 37 C.F.R. § 1.132

ASSISTANT COMMISSIONER FOR PATENTS
WASHINGTON, D.C. 20231

SIR:

Now comes, Kensaku KOMATSU, a citizen of Japan, residing at 1621, Sakazu, Kurashiki-Shi, Okayama 710-8622, Japan, who deposes and states:

1) I am a co-inventor in the above-identified application.

2) I graduated from Faculty of Technology, Tokyo University of Agriculture and Technology in March, 1988.

3) I entered Kuraray Co., Ltd, in April, 1988, and

4) I have been conducting research and development in the development of membranes.

5) The following experiments were conducted by me or under my direct supervision and control.

5.1 Experiments

Example 1

Example 1 of the present application was repeated. A spinning dope having 20 wt% of polysulfone (UDEL-P1800, manufactured by and available from AMOCO Japan, Ltd. Hereinafter, this particular polysulfone is referred to as PSf.), 6 wt% of ethylene glycol (hereinafter, referred to as EG), 18 wt% of silicone oxide having an average particle size of 4.5 μm, and 54 wt% of N,N-dimethyl acetamide (hereinafter, referred to as DMAc) was prepared by the following procedure. Specifically, after EG was dissolved into DMAc, the silica powder was uniformly dispersed in the DMAc solution using a home jettor to provide a dispersed liquid which was subsequently added to PSf. The resultant mixture was then stirred for 8 hours at 60℃ to thereby dissolve the PSf, resulting in the white colored slurry in which the silicon oxide was uniformly dispersed. This slurry was used as the spinning dope.

The spinning dope obtained in the manner described above was thereafter degassed. While the degassed spinning dope was maintained at 50℃, the degassed spinning dope was discharged from a nozzle of a double ring structure, 1.65 mm in outer diameter and 0.8 mm in inner diameter, at 50℃ together with the coagulation liquid containing 80 wt% of N,N-dimethyl formamide (hereinafter referred to as DMF), 19wt% of water and 1 wt% of polyvinyl alcohol (PVA205, manufactured by and available from KURARAY CO., Ltd. This particular polyvinyl alcohol is hereinafter referred to as PVA.). After the discharged spinning dope had been advanced 10 cm within a dry zone at a temperature of 50℃ and a relative humidity of 90%, it was introduced into water of 50℃ which formed a coagulation bath, to thereby provide hollow fiber

membranes. The spinning speed was set to 4.5 m/min.

Subsequently, the resultant hollow fiber membranes were washed with hot water of 98℃ for two hours to extract DMAc, EG and PVA therefrom. Thereafter, the hollow fiber membranes were immersed for one hour in an aqueous solution heated to 60℃ and containing glutaraldehyde in a proportion of 3g/L and sulfuric acid in a proportion of 30 g/L to cross-link the PVA. The hollow fiber membranes were then immersed for two hours in an aqueous solution heated to 80℃ and containing 13 wt% of sodium hydroxide to extract and remove silicon oxide contained therein. Also, the hollow fiber membranes were then washed for 2 hours with hot water of 90℃, followed by drying at 45℃ for 16 hours or more to thereby complete the hollow fiber membranes of 1.3 mm in outer diameter and 0.8 mm in inner diameter.

The resultant hollow fiber membranes were found to have a pure water permeate flow of 135,000 L/m^2/hr/100kPa and a particle cutoff of 2.4 μm. The term "pure water permeate flow" was measured in the following manner. Specifically, using a single-open ended hollow fiber membrane module having an effective length of 3 cm, and using pure water as untreated water, the amount of the pure water permeated per unitary time when filtered(extra-pressurized filtration) from outside to inside of the hollow fiber membranes under a filtering pressure of 50 kPa at a temperature of 25 ℃ was measured. A numerical value converted into the amount of pure water permeate per unitary membrane area, unitary time and unitary pressure represented the pure water permeate flow.

Also, the term "particle cutoff" referred hereinbefore and hereinafter is intended to mean the particle size (S) of particles with which the hollow fiber membrane can exhibit a blocking rate of a 90%. This particle cutoff can be determined by measuring the blocking rates of two kinds of particles having different particle sizes and determining the value S at which in the following approximate equation (1) the parameter R (i.e., the blocking rate) can attain 90.

$$R = 100/(1-M\times\exp(-A\times\log(S)))$$

wherein A and M represent a constant determined by the hollow fiber membrane and are calculated based on two or more measured values of the blocking rates.

Electron microphotographs showing an outer surface, an inner surface and a section of the resultant hollow fiber membranes are shown in Figs.1,2 and 3, respectively, which are attached to and incorporated into this Declaration.

Example 2

Example 1 was repeated except that 6 wt% of EG was substituted with the same amount of DMAc in the spinning dope.

The resultant hollow fiber membranes were found to have a pure water permeate flow of 985 $L/m^2/hr/100kPa$ and a particle cutoff of 0.9μm. Electron microphotographs showing an outer surface, an inner surface and a section of the resultant hollow fiber membranes are shown in Figs.4,5 and 6, respectively, which are attached to and incorporated into this Declaration.

Example 3

Example 2 of the present application was repeated. The hollow fiber membranes were manufactured in a manner similar to that in Example 1 of the present application, except that a spinning dope containing 20 wt% of PSf, 6 wt% of EG, 20 wt% of silicon oxide having an average particle size of 11μm, 2 wt% of silicon oxide having an average particle size of 4.5μm and 52 wt% of DMAc, was used. The resultant hollow fiber membranes were found to have a pure water permeate flow of 520,000 L/m^2/hr/100kPa and a particle cutoff of 5.0μm. Electron microphotographs showing an outer surface, an inner surface and a section of the resultant hollow fiber membranes are shown in Figs.7,8 and 9, respectively, which are attached to and incorporated into this Declaration.

Example 4

Example 3 was repeated except that 6 wt% of EG was substituted with the same amount of DMAc in the spinning dope. The resultant hollow fiber membranes were found to have a pure water permeate flow of 1,746 L/m^2/hr/100kPa and a particle cutoff of 2.1μm. Electron microphotographs showing an outer surface, an inner surface and a section of the resultant hollow fiber membranes are shown in Figs.10,11 and 12, respectively, which are attached to and incorporated into this Declaration.

Example 5

Example 3 of the present application was repeated in a manner similar to that in Example 1 of the present application, except that a spinning dope containing 20 wt% of PSf, 4 wt% of EG, 14 wt% of silicon oxide having an average particle size of 1.5 μm and 62 wt% of DMF was used to manufacture the hollow fiber membranes. The resultant hollow fiber membranes were found to have a pure water permeate flow of 39,000 L/m^2/hr/100kPa and a particle cutoff of 1.2μm. Electron microphotographs showing an outer surface, an inner surface and a section of the resultant hollow fiber membranes are shown in Figs.13,14 and 15, respectively, which are attached to and incorporated into this Declaration.

Example 6

Example 5 was repeated except that 4 wt% of EG was substituted with the same amount of DMF in the spinning dope. The resultant hollow fiber membranes were found to have a pure water permeate flow of 5,567 L/m^2/hr/100kPa and a particle cutoff of 0.9μm. Electron microphotographs showing an outer surface, an inner surface and a section of the resultant hollow fiber membranes are shown in Figs.16,17 and 18, respectively, which are attached to and incorporated into this Declaration.

5.2 Results

	Example 1		Example 2	
spinning dope	polysulfone	20wt%	polysulfone	20wt%
	ethylene glycol	6wt%	ethylene glycol	—
	silicone oxide (4.5 μm)	18wt%	silicone oxide (4.5 μm)	18wt%
	N,N-dimethyl acetoamide	54wt%	N,N-dimethyl acetoamide	60wt%
pure water permeate flow	135000 L/m²/hr/100KPa		985 L/m²/hr/100KPa	
particle cutoff	2.4 μm		0.9 μm	

	Example 3		Example 4	
spinning dope	polysulfone	20wt%	polysulfone	20wt%
	ethylene glycol	6wt%	ethylene glycol	—
	silicone oxide (11 μm)	20wt%	silicone oxide (11 μm)	20wt%
	silicone oxide (4.5 μm)	2wt%	silicone oxide (4.5 μm)	2wt%
	N,N-dimethyl acetoamide	52wt%	N,N-dimethyl acetoamide	58wt%
pure water permeate flow	520000 L/m²/hr/100KPa		1746 L/m²/hr/100KPa	
particle cutoff	5.0 μm		2.1 μm	

	Example 5		Example 6	
spinning dope	polysulfone	20wt%	polysulfone	20wt%
	ethylene glycol	4wt%	ethylene glycol	—
	silicone oxide (1.5 μm)	14wt%	silicone oxide (1.5 μm)	14wt%
	N,N-dimethyl acetoamide	62wt%	N,N-dimethyl acetoamide	66wt%
pure water permeate flow	39000 L/m²/hr/100KPa		5567 L/m²/hr/100KPa	
particle cutoff	1.2 μm		0.9 μm	

6) From the above results, it is clear that the additive in the present invention contributes greatly in obtaining large amount of a pure water permeate flow and large amount of a particle cutoff. This is clearly unexpected.

7) Hence, I am of the opinion that the present invention and the results thereof are unexpected, important and commercially significant.

8) I further declare that all statements made on information and belief are believed to be true; and further that these statements were made with the knowledge that willful false statements and the like so made are punishable by fine or imprisonment, or both, under section 1001 of Title 18 of the United States Code and that such willful false statements may jeopardize the validity of this application or any patent issuing thereon.

9) Further deponent saith not.

Kensaku Komatsu

Name

Kensaku Komatsu

Signature

November 17, 2003

Date